

## PCT

## INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference <b>6900-198 MIS</b>	<b>FOR FURTHER ACTION</b> see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. <b>PCT/CA 99/ 00155</b>	International filing date (day, month, year) <b>22/02/1999</b>	(Earliest) Priority Date (day, month, year) <b>23/02/1998</b>
Applicant <b>POLYPHALT INC. et al.</b>		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 2 sheets.



It is also accompanied by a copy of each prior art document cited in this report.

## 1. Basis of the report

- a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.



the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

- b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing:



contained in the international application in written form.



filed together with the international application in computer readable form.



furnished subsequently to this Authority in written form.



furnished subsequently to this Authority in computer readable form.



the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.



the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ **Certain claims were found unsearchable** (See Box I).

3. ☐ **Unity of invention is lacking** (see Box II).

4. With regard to the **title**,



the text is approved as submitted by the applicant.



the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,



the text is approved as submitted by the applicant.



the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No.



as suggested by the applicant.



because the applicant failed to suggest a figure.



because this figure better characterizes the invention.



None of the figures.

# INTERNATIONAL SEARCH REPORT

National Application No.  
PCT/CA 99/00155

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C08L95/00 D21C11/00 //(C08L95/00,97:00)

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C08L D21C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3 956 002 A (MOORER HOWARD H) 11 May 1976 see column 2, line 3-11	
A	US 4 293 459 A (DETROIT WILLIAM J) 6 October 1981 see claim 1	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

23 June 1999

Date of mailing of the international search report

29/06/1999

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl.  
Fax (+31-70) 340-3016

Authorized officer

Leroy, A

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/CA 99/00155

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 3956002	A	11-05-1976	US 4088505 A	09-05-1978
US 4293459	A	06-10-1981	NONE	

16  
REC'D 02 JUN 2000

WIPO

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 6900-198 MIS	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/CA99/00155	International filing date (day/month/year) 22/02/1999	Priority date (day/month/year) 23/02/1998
International Patent Classification (IPC) or national classification and IPC C08L95/00		
Applicant POLYPHALT INC. et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.



2. This REPORT consists of a total of 5 sheets, including this cover sheet.

- ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 4 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☒ Certain observations on the international application

Date of submission of the demand  10/09/1999	Date of completion of this report  30.05.2000
Name and mailing address of the international preliminary examining authority:   European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer  Idez, C  Telephone No. +49 89 2399 8665  

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/CA99/00155

**I. Basis of the report**

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

**Description, pages:**

1-3,7-14 as originally filed  
4-6,6a with telefax of 23/02/2000

**Claims, No.:**

1-31 as originally filed

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:  
☐ the claims, Nos.:  
☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

**1. Statement**

Novelty (N)	Yes:	Claims	7-31
	No:	Claims	1-6
Inventive step (IS)	Yes:	Claims	7-31
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-31
	No:	Claims	

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/CA99/00155

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2. Citations and explanations

**see separate sheet**

**VIII. Certain observations on the international application**

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

**see separate sheet**

1) Concerning point V:

1.1) D2 discloses bituminous compositions comprising a dewatered lignin containing spent alkaline pulping liquor.

The arguments of the Applicant that in D2 this component is not "stably" dispersed in the bitumen cannot be presently be accepted, since it is unclear what is exactly meant by the wording "stably dispersed". In that respect, it should also be noted that the tests used for checking the "stability" of the compositions according to the present application show that a separation of the components occurs ( see for example table 3, compositions AC-B-19, AC-B-23, AC-B-20) .

1.2) The arguments of the Applicant stating that D2 does refer to "black liquor" can only be relevant provided the chemical composition of the dewatered "black liquor" ( see present claim 6) is different from the composition of the dewatered spent alkaline pulping liquor according to D2.

1.3) Furthermore unclear wordings ( stably dispersed, black liquor) cannot be used to distinguish the claimed subject-matter from the prior art.

1.4) Thus, D2 is presently considered as a novelty destroying document for the subject-matter of present claims 1-6. (Art.33(2)).

1.5) The subject-matter of present claims 8 to 31 is presently considered as novel and inventive, since there is no indication in the cited documents that bituminous compositions showing an acceptable storage stability ( low tendency to phase separation) can be obtained by the claimed processes. (Art.33(2) and 33(3)).

2) Concerning point VIII:

2.1) According to the Applicant the wording "stably dispersed" has a limiting function. The mention of this property in the claims without indicating the method and

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

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International application No. PCT/CA99/00155

criteria according to which the dewatered pulping liquor is considered as "stably dispersed" in the claimed compositions, renders the claims 1-6 unclear. (Art.6).

2.2) It is unclear which is the difference of subject-matter between independent claim 16 and present claim 12 ( dependent on claim 7), since the process steps of independent 16 exactly correspond to the process steps of the combination of claim 12 with claim 7 on which it depends. The same applies for claims 17 and 13. (Art 6).

2.3) The wordings "substantially" , "about", "elevated temperature" are vague and render unclear the claims in which they occur. (Art.6).

2.4) The wording "black liquor" is vague and does clearly define the matter for which protection is sought. This renders the claims in which this wording occurs unclear. (Art.6).



procedure involves essentially two steps. In the first step, the black liquor is converted from its aqueous colloidal system to an anhydrous colloidal dispersion in a lubricating oil medium in which a surfactant, which  
5 may be an anionic surfactant, such as DDBSA (dodecylbenzene sulfuric acid), is used to promote the provision of the anhydrous colloidal dispersion. The process may be effected under low shear conditions.

The anhydrous colloidal dispersion of lignin in oil  
10 may be mixed, in a second step, with asphalt bitumen, producing a unique combination, which is a novel bituminous composition.

In another embodiment of the invention, the procedure again involves essentially two steps. First,  
15 bitumen is treated with an inorganic acid, such as sulfuric acid, at elevated temperature to provide an acid-treated bitumen. The black alkaline liquor is then added slowly to the acid-treated bitumen under agitation for de-watering the black liquor at a controlled rate to  
20 form a stable, substantially anhydrous, colloidal dispersion of lignin in the bitumen, also to provide a novel bituminous composition.

#### DETAILED DESCRIPTION OF THE INVENTION

Referring first to the first embodiment of the  
25 invention, a lubricating oil is mixed with black liquor at temperatures below the water boiling point. A surfactant, which may be DDBSA (dodecylbenzene sulfuric acid), is selected to be added to the mixture to facilitate the colloidal dispersion of black liquor in  
30 the lubricating oil while de-watering is gradually carried out at an elevated temperature. The resulting cream-like paste product is miscible with hot liquid asphalt at any ratio to form a novel bituminous composition.

35 The lubricating oil used in the first embodiment of the invention may be a re-refined waste motor oil or

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APT 34 AMDT

other convenient petroleum-based oil. Contaminants of waste motor oil, such as dirt, lead, arsenic and other harmful metals and chemicals, are removed from the waste motor oil to produce a clean base lubricating oil by the re-refining process.

The lubricating oil may be a selected fraction of refined mineral oil used for lubrication of moving surfaces. Such fraction may range in consistency from thin liquid to grease-like substances. Usually, lubricating oils contain small amounts of additives to impart special properties, such as viscosity index and detergency.

The surfactant or dispersing agent used in the process may typically be dodecylbenzene sulfonic acid (DDBSA), although other anionic surfactants may be employed, if desired, such as fatty acid, linear alkyl sulfonates having 10 or more carbon atoms in the chain. DDBSA is benzene with dodecene, and the resulting dodecylbenzene is sulfonated. DDBSA may be neutralized with caustic soda from the alkaline black liquor or other convenient source to promote the detergency of the lubricating oil and to reduce surface tension in the mixture.

However, a surfactant need not always be necessary, if the lubricating oil employed has a detergency high enough to ensure that a colloidal dispersion of the solid residue from the black liquor is achieved in the lubricating oil medium.

The lignin-oil dispersion produced by either of these procedures may be smoothly blended with bitumen to provide the desired lignin-asphalt composition.

Referring now to the second embodiment of the invention, the bitumen first is treated with a strong mineral acid, such as sulfuric acid, at elevated temperature and then the alkaline black liquor is added to the treated bitumen at a controlled rate under

REF ID: A61111

agitation to effect de-watering of the black liquor to form a stable, substantially anhydrous, colloidal dispersion in the bitumen.

5 An inorganic acid is contacted with, or added to, the asphalt to form an acid treated asphalt. In general, the acid addition shifts the asphalt structure from a sol to a gel, lowers the temperature susceptibility of the asphalt and improves the stability of the additive dispersion in the treated asphalt. Since black liquor  
10 is an aqueous alkaline system, it is critical that the acid not be added to the asphalt after addition of or with the black liquor.

Preferably, the acid is added slowly to the asphalt to avoid foaming, which may occur if all the acid were  
15 added at one time. The inorganic acid content of the asphalt resulting from the acid treatment is not critical, but normally is in the range between about 0.2 and about 3.5 wt%, preferably between about 0.5 and about 2.5 wt% of the asphalt. Although a wide variety  
20 of inorganic acids can be used for treatment of the asphalt, the inorganic acid is preferably selected from the group consisting of sulfuric acid, phosphoric acid, poly-phosphoric acid, phosphorous pentoxide, hydrochloric acid, and mixtures thereof. The sulfuric  
25 acid, phosphoric acid or poly-phosphoric acid are preferred inorganic acids, with the sulfuric acid being particularly preferred.

Following acid addition, the alkaline black liquor is then added slowly to the acid-treated bitumen at an  
30 elevated temperature, typically around 100°C, under agitation for a certain period of time to permit de-watering of the black liquor to occur while the solids are incorporated into the bitumen. The temperature of the final composition next is increased above the  
35 boiling point of water, typically up to around 160°C until residual water is completely evaporated off to

## PATENT COOPERATION TREATY

PCT

## NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents  
United States Patent and Trademark  
Office  
Box PCT  
Washington, D.C. 20231  
ÉTATS-UNIS D'AMÉRIQUE

in its capacity as elected Office

<b>Date of mailing</b> (day month/year) 02 November 1999 (02.11.99)	
<b>International application No.</b> PCT/CA99/00155	<b>Applicant's or agent's file reference</b> 6900-198 MIS
<b>International filing date</b> (day/month/year) 22 February 1999 (22.02.99)	<b>Priority date</b> (day/month/year) 23 February 1998 (23.02.98)
<b>Applicant</b> LIANG, Zhi-Zhong	

1. The designated Office is hereby notified of its election made:



in the demand filed with the International Preliminary Examining Authority on:

10 September 1999 (10.09.99)



in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was

was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer C. Carrié Telephone No.: (41-22) 338.83.38
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**RECEIVED**

JUN 6 2000

**PCT****SIM, HUGHES, ASHTON & McK**From the  
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

STEWART, Michael  
Sim & McBumey  
330 University Avenue  
6th floor  
Toronto, Ontario M5G 1R7  
CANADANOTIFICATION OF TRANSMITTAL OF  
THE INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT  
(PCT Rule 71.1)Date of mailing  
(day/month/year) 30.05.2000Applicant's or agent's file reference  
6900-198 MIS**IMPORTANT NOTIFICATION**International application No.  
PCT/CA99/00155International filing date (day/month/year)  
22/02/1999Priority date (day/month/year)  
23/02/1998Applicant  
POLYPHALT INC. et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

**4. REMINDER**

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/



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D-80298 Munich  
Tel. +49 89 2399 - 0 Tx: 523656 epmu d  
Fax: +49 89 2399 - 4465

Authorized officer

Hardy Magliano, N

Tel +49 89 2399-8151



# PATENT COOPERATION TREATY

## PCT

### INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 6900-198 MIS	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/CA99/00155	International filing date ( <i>day/month/year</i> ) 22/02/1999	Priority date ( <i>day/month/year</i> ) 23/02/1998
International Patent Classification (IPC) or national classification and IPC C08L95/00		
Applicant POLYPHALT INC. et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

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- ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 4 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☒ Certain observations on the international application

Date of submission of the demand  10/09/1999	Date of completion of this report  30.05.2000
Name and mailing address of the international preliminary examining authority:  <div style="display: flex; align-items: center;"> <div>             European Patent Office              D-80298 Munich              Tel. +49 89 2399 - 0 Tx: 523656 epmu d              Fax: +49 89 2399 - 4465           </div> </div>	Authorized officer  Idez, C  Telephone No. +49 89 2399 8665



**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/CA99/00155

**I. Basis of the report**

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

**Description, pages:**

1-3,7-14 as originally filed  
4-6,6a with telefax of 23/02/2000

**Claims, No.:**

1-31 as originally filed

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:  
☐ the claims, Nos.:  
☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

**1. Statement**

Novelty (N)	Yes: Claims 7-31
	No: Claims 1-6
Inventive step (IS)	Yes: Claims 7-31
	No: Claims
Industrial applicability (IA)	Yes: Claims 1-31
	No: Claims

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/CA99/00155

---

2. Citations and explanations

**see separate sheet**

**VIII. Certain observations on the international application**

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

**see separate sheet**



**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

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International application No. PCT/CA99/00155

1) Concerning point V:

1.1) D2 discloses bituminous compositions comprising a dewatered lignin containing spent alkaline pulping liquor.

The arguments of the Applicant that in D2 this component is not "stably" dispersed in the bitumen cannot be presently be accepted, since it is unclear what is exactly meant by the wording "stably dispersed". In that respect, it should also be noted that the tests used for checking the "stability" of the compositions according to the present application show that a separation of the components occurs ( see for example table 3, compositions AC-B-19, AC-B-23, AC-B-20) .

1.2) The arguments of the Applicant stating that D2 does refer to "black liquor" can only be relevant provided the chemical composition of the dewatered "black liquor" ( see present claim 6) is different from the composition of the dewatered spent alkaline pulping liquor according to D2.

1.3) Furthermore unclear wordings ( stably dispersed, black liquor) cannot be used to distinguish the claimed subject-matter from the prior art.

1.4) Thus, D2 is presently considered as a novelty destroying document for the subject-matter of present claims 1-6. (Art.33(2)).

1.5) The subject-matter of present claims 8 to 31 is presently considered as novel and inventive, since there is no indication in the cited documents that bituminous compositions showing an acceptable storage stability ( low tendency to phase separation) can be obtained by the claimed processes. (Art.33(2) and 33(3)).

2) Concerning point VIII:

2.1) According to the Applicant the wording "stably dispersed" has a limiting function. The mention of this property in the claims without indicating the method and

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

---

International application No. PCT/CA99/00155

criteria according to which the dewatered pulping liquor is considered as "stably dispersed" in the claimed compositions, renders the claims 1-6 unclear. (Art.6).

2.2) It is unclear which is the difference of subject-matter between independent claim 16 and present claim 12 (dependent on claim 7), since the process steps of independent 16 exactly correspond to the process steps of the combination of claim 12 with claim 7 on which it depends. The same applies for claims 17 and 13. (Art 6).

2.3) The wordings "substantially", "about", "elevated temperature" are vague and render unclear the claims in which they occur. (Art.6).

2.4) The wording "black liquor" is vague and does not clearly define the matter for which protection is sought. This renders the claims in which this wording occurs unclear. (Art.6).

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528 Recd 23 AUG 2000

4

procedure involves essentially two steps. In the first step, the black liquor is converted from its aqueous colloidal system to an anhydrous colloidal dispersion in a lubricating oil medium in which a surfactant, which  
5 may be an anionic surfactant, such as DDBSA (dodecylbenzene sulfuric acid), is used to promote the provision of the anhydrous colloidal dispersion. The process may be effected under low shear conditions.

10 The anhydrous colloidal dispersion of lignin in oil may be mixed, in a second step, with asphalt bitumen, producing a unique combination, which is a novel bituminous composition.

15 In another embodiment of the invention, the procedure again involves essentially two steps. First, bitumen is treated with an inorganic acid, such as sulfuric acid, at elevated temperature to provide an acid-treated bitumen. The black alkaline liquor is then added slowly to the acid-treated bitumen under agitation for de-watering the black liquor at a controlled rate to  
20 form a stable, substantially anhydrous, colloidal dispersion of lignin in the bitumen, also to provide a novel bituminous composition.

25 Accordingly, in another aspect of the present invention, there is provided a bituminous composition comprising bitumen, and dewatered lignin-containing spent alkaline pulping liquor dispersed therein. The dewatered spent alkaline pulping liquor may be present in an amount up to about 50 wt.% of the bitumen, preferably about 5 to about 25 wt.% of the bitumen.

30 In such composition, the dewatered spent alkaline pulping liquor may comprise an anhydrous colloidal dispersion of lignin in a lubricating oil. The dewatered spent alkaline pulping liquor may comprise a stable, substantially anhydrous, colloidal dispersion of  
35 lignin in the bitumen.

DETAILED DESCRIPTION OF THE INVENTION

Referring first to the first embodiment of the invention, a lubricating oil is mixed with black liquor at temperatures below the water boiling point. A  
5 surfactant, which may be DDBSA (dodecylbenzene sulfuric acid), is selected to be added to the mixture to facilitate the colloidal dispersion of black liquor in the lubricating oil while de-watering is gradually carried out at an elevated temperature. The dewatering  
10 may be effected by starting at an elevated temperature, which may be about 100° to about 180°C, preferably about 110° to about 160°C. The resulting cream-like paste product is miscible with hot liquid asphalt at any ratio to form a novel bituminous composition.

15 The lubricating oil used in the first embodiment of the invention may be a re-refined waste motor oil or other convenient petroleum-based oil. Contaminants of waste motor oil, such as dirt, lead, arsenic and other harmful metals and chemicals, are removed from the waste  
20 motor oil to produce a clean base lubricating oil by the re-refining process.

The lubricating oil may be a selected fraction of refined mineral oil used for lubrication of moving  
25 surfaces. Such fraction may range in consistency from thin liquid to grease-like substances. Usually, lubricating oils contain small amounts of additives to impart special properties, such as viscosity index and detergency.

The surfactant or dispersing agent used in the  
30 process may typically be dodecylbenzene sulfuric acid (DDBSA), although other anionic surfactants may be employed, if desired, such as fatty acid, linear alkyl sulfonates having 10 or more carbon atoms in the chain. DDBSA is benzene with dodecene, and the resulting  
35 dodecylbenzene is sulfonated. DDBSA may be neutralized with caustic soda from the alkaline black liquor or

other convenient source to promote the detergency of the lubricating oil and to reduce surface tension in the mixture.

5 However, a surfactant need not always be necessary, if the lubricating oil employed has a detergency high enough to ensure that a colloidal dispersion of the solid residue from the black liquor is achieved in the lubricating oil medium.

10 The lignin-oil dispersion produced by either of these procedures may be smoothly blended with bitumen to provide the desired lignin-asphalt composition. The spent pulping liquor, which may be black liquor from a kraft pulp mill, may be present in an amount of about 25 to about 60 wt.% of the composition, preferably about 25  
15 to 40 wt.% of the composition.

Referring now to the second embodiment of the invention, the bitumen first is treated with a strong mineral acid, such as sulfuric acid, at elevated temperature and then the alkaline black liquor is added  
20 to the treated bitumen at a controlled rate under agitation to effect de-watering of the black liquor to form a stable, substantially anhydrous, colloidal dispersion in the bitumen. The elevated temperature may be about 100° to about 180°C, preferably about 110° to  
25 about 160°C. The spent pulping liquor may be present in an amount of about 5 to about 50 wt.% of the composition, preferably about 10 to about 30 wt.%

An inorganic acid is contacted with, or added to, the asphalt to form an acid treated asphalt. In general,  
30 the acid addition shifts the asphalt structure from a sol to a gel, lowers the temperature susceptibility of the asphalt and improves the stability of the additive dispersion in the treated asphalt. Since black liquor is an aqueous alkaline system, it is critical that the  
35 acid not be added to the asphalt after addition of or with the black liquor.

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Preferably, the acid is added slowly to the asphalt to avoid foaming, which may occur if all the acid were added at one time. The inorganic acid content of the asphalt resulting from the acid treatment is not  
5 critical, but normally is in the range between about 0.2 and about 3.5 wt%, preferably between about 0.5 and about 2.5 wt% of the asphalt. Although a wide variety of inorganic acids can be used for treatment of the asphalt, the inorganic acid is preferably selected from  
10 the group consisting of sulfuric acid, phosphoric acid, poly-phosphoric acid, phosphorous pentoxide, hydrochloric acid, and mixtures thereof. The sulfuric acid, phosphoric acid or poly-phosphoric acid are preferred inorganic acids, with the sulfuric acid being  
15 particularly preferred.

Following acid addition, the alkaline black liquor is then added slowly to the acid-treated bitumen at an elevated temperature, typically around 100°C, under agitation for a certain period of time to permit de-  
20 watering of the black liquor to occur while the solids are incorporated into the bitumen. The temperature of the final composition next is increased above the boiling point of water, typically up to around 160°C until residual water is completely evaporated off to